are consistently high, the measured density is high, the magnetic susceptibility is high, and the electrical resistivity indicates metallic behavior instead of the expected semiconducting behavior which is found for monoclinic (D type)  $Dy_2S_3$ . Of course, this does not preclude the possibility that more stoichiometric cubic  $Dy_2S_3$  might be prepared under other conditions.

The unit cell dimensions of the Th<sub>3</sub>P<sub>4</sub> type rare earth sulfides<sup>2</sup> are nearly invariant over the composition range  $R_{2.677}S_4$  ( $R_2S_3$ ) to  $R_3S_4$  for R = La, Ce, Pr, and Nd. This means that the average cation-to-sulfur distance is nearly invariant over this range because the *average* distance changes very little with changes in the one positional parameter. The average cation size might be expected to increase since formally some cations are being reduced from  $R^{3+}$  to  $R^{2+}$ . The filling of some cation vacancies would not be expected to compensate for such an increase; consequently, the best rationalization appears to be that the cations do not increase in size when they are formally reduced because the additional electrons are not localized at the cations. The expected increase in unit cell dimensions does occur

on going from  $\text{Sm}_2\text{S}_3$  to  $\text{Sm}_3\text{S}_4$  indicating that  $\text{Sm}^{2+}$  is really present and the additional electron has not been delocalized. This conclusion is supported by magnetic and electrical data<sup>24</sup> in that CeS is metallic with a magnetic moment corresponding to Ce<sup>3+</sup>, whereas SmS is a semiconductor with a magnetic moment corresponding to Sm<sup>2+</sup>. Of course, Sm<sup>2+</sup> is well known in other compounds, whereas Ce<sup>2+</sup> is not.

At this time it is not possible to state with certainty whether or not the  $Th_3P_4$  phase can exist at *exactly* the sesquisulfide stoichiometry for the rare earth sulfides. It is possible that this C-type structure is a valid high-temperature form, but it is likewise possible that in the high-temperature preparations of this form at least some sulfur is always lost. Clarification of this point will have to await more sophisticated preparative or analytical techniques.

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# The Crystal Structure of a Brominated Carborane–Metal Sandwich Compound, N(CH<sub>3</sub>)<sub>4</sub>[(B<sub>9</sub>C<sub>2</sub>H<sub>8</sub>Br<sub>3</sub>)<sub>2</sub>Co]<sup>1</sup>

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The crystal structure of the tetramethylammonium salt of bis- $\pi$ -(5,9,10-tribromo-(1)-2,3-dicarbollyl)cobalt(III), N(CH<sub>3</sub>)<sub>4</sub>-[(B<sub>3</sub>C<sub>2</sub>H<sub>8</sub>Br<sub>3</sub>)<sub>2</sub>Co], has been determined by an X-ray diffraction study of a single-crystal specimen. The monoclinic unit cell, space group P2<sub>1</sub>/c, with  $a = 19.893 \pm 0.010$  Å,  $b = 19.487 \pm 0.010$  Å,  $c = 15.058 \pm 0.010$  Å, and  $\beta = 93.15 \pm 0.05^{\circ}$ , contains eight formula units and four crystallographically independent anions. The calculated density is 1.967 g/cm<sup>3</sup>, in agreement with the measured value of  $1.98 \pm 0.01$  g/cm<sup>3</sup>. The structure was solved by statistical methods and refined by a least-squares procedure to a conventional R of 8.7% on 3002 data collected by counter methods. All four independent anions have the same shape to within the accuracy of this determination. The anion, the product of a bromination during which the bis(dicarbollyl)cobalt "sandwich" is believed to remain intact, consists of two substituted **ico**sahedra with the cobalt as their common vertex. In each icosahedron, the carbons are adjacent to each other and to the cobalt, while the three borons bonded to bromine form the corners of a triangular face. Two corners of this face are as far as possible from the carbons, and the third is adjacent to the cobalt. These bromination sites are consistent with a charge distribution in the reactant which is analogous to that in o-B1<sub>0</sub>C<sub>2</sub>H<sub>12</sub> but modified slightly by the presence of the Co(III).

### Introduction

Hawthorne and coworkers<sup>3-8</sup> have recently synthesized a number of  $\pi$ -dicarbollylmetal compounds analogous to the  $\pi$ -cyclopentadienyl "sandwich" compounds. They found<sup>8,9</sup> that one of these substances,  $Co(B_9C_2H_{11})_2^-$ , could be electrophilically brominated by treatment with neat bromine or bromine in glacial acetic acid to give  $Co(B_9C_2H_8Br_3)_2^-$ . This is thought<sup>9</sup> to be the first example of a substitution upon the intact bis(dicarbollyl)metal ''sandwich'' compound.

This determination of the crystal structure of the tetramethylammonium salt of the product ion once again establishes the bis(dicarbollyl)metal structure as two icosahedra with the metal atom as their common vertex. This work was undertaken in order to ascer-

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<sup>(3)</sup> M. F. Hawthorne, D. C. Young, and P. A. Wegner, J. Am. Chem. Soc., 87, 1818 (1965).

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(8)</sup> M. F. Hawthorne, et al., ibid., 90, 879 (1968).

<sup>(9)</sup> M. F. Hawthorne, private communication.

tain the positions of bromine substitution upon these icosahedra. It was found that the bromines are bonded to three borons in each icosahedron which form a triangular face, one corner of which is adjacent to the cobalt atom. The other two corners of the brominated face are as far as possible from the two carbon atoms, which are adjacent to each other and to the cobalt.

## **Experimental Section**

Crystals of  $N(CH_3)_4[(B_8C_2H_8Br_3)_2C_0]$  were sent to us by Professor M. F. Hawthorne of the University of California, Riverside, Calif. Intensity data were collected from two of these orange-brown crystals, both of which were approximately 0.1 mm square and 0.05 mm thick. The calculated linear absorption coefficient,  $\mu$ , was 153 cm<sup>-1</sup> (for Cu K $\alpha$ ). We estimate that the absorption effect on intensities may vary by a factor of the order of 1.5 between the most extreme cases. Both crystals were mounted by being glued to the ends of Pyrex glass fibers in such a way that the fiber, the instrument  $\varphi$  axis, and the b axis were all perpendicular to the plate. A General Electric XRD-5 X-ray diffractometer equipped with a molybdenum X-ray tube, a manual quarter-circle Eulerian-cradle goniostat, and a Zr filter at the receiving slit was used to measure cell dimensions. They were obtained from carefully measured  $2\theta$  ( $\theta$  is the Bragg angle) values of the h00, 0k0, and 00l reflections. The  $\alpha$  doublet was resolved ( $\lambda$ (Mo K $\alpha_1$ ) 0.70926 Å). The  $\beta$  angle was obtained directly from the angle on the  $\varphi$  circle between the h00 and 00l sets of reflections. A card-controlled version of the same apparatus, using a copper X-ray tube and a Ni filter between crystal and counter, measured intensities by scanning  $2\theta$  at the rate of  $1^{\circ}/\text{min},$  beginning 0.7° below the  $\alpha_1$  peak and ending 0.7° above the  $\alpha_2$  peak ( $\lambda$ (Cu K $\alpha_1$ , K $\alpha_2$ ) 1.5405, 1.5443 Å). The takeoff angle at the X-ray tube anode was approximately 2°. Coincidence losses were negligible. Ten-second stationary background counts were taken at each end of the scan. Three reflections which were measured at frequent intervals showed no systematic trends over the course of taking either data set. The maximum  $2\theta$  was  $75^\circ$  ( $\lambda^{-1}\sin\theta = 0.395$ ).

The first crystal yielded 1598 independent intensities before it was accidentally destroyed. Of these,  $\sim 23\%$  were measured once,  $\sim 64\%$  either two or three times, and the remainder up to six times. These multiple measurements include remeasurements of the same reflection and measurements of equivalent  $(\hbar k \bar{l})$  reflections in an irregular pattern caused by the fact that these measurements were made during an initial exploration of the use of the newly acquired diffractometer. The second crystal gave a complete set of 3002 independent intensities. Approximately 75% of these (those with  $|l| \leq 6$ ) also had their equivalent  $(\hbar k \bar{l})$  reflections measured. These figures do not include the space group extinctions which were also measured and found to confirm the extinction rules  $(0k0, k \neq 2n, \text{ and } h0l, l \neq 2n)$  obtained from preliminary Weissenberg photographs.

The intensity, I, and standard deviation,  $\sigma(I)$ , for each measurement were calculated by:  $I = C - (t_c/2t_B)(B_1 + B_2)$  and  $\sigma^2(I) = C + (t_C/2t_B)^2(B_1 + B_2) + (0.06I)^2$ , where C is the number of counts accumulated in scanning through the reflection in time  $t_c$ , and  $B_1$  and  $B_2$  are the background counts, each obtained in time  $t_B$ . Within each data set, multiple measurements (including measurements of equivalent reflections) were averaged and the averages were assigned standard deviations equal to the greater of:  $(\Sigma_i \sigma_i^2/n^2)^{1/2}$  or  $(\Sigma_i \Delta_i^2/n^2)^{1/2}$ , where  $\sigma_i$ and  $\Delta_i$  are the standard deviation and deviation from the average of the ith measurement and n is the number of measurements averaged. The latter expression did give the greater value in 10% of the cases in the first set and 15% in the second set. Later, it was realized that this procedure tends to "average out" the 0.06 I included in  $\sigma(I)$ , but this was approximately corrected when the two data sets were combined as described below. Observed structure factors for reflections which were included in both data sets were larger in the first set about as often as in the

second, and corresponding values were typically in disagreement by 5%. There was no statistically valid deviation from equal scaling, and the combined data set was generated by averaging (without scaling) those  $F_{\rm o}$  values<sup>10</sup> which the two sets had in common and setting

$$\sigma^{2}(F_{av}) = 0.5[\sigma^{2}(F_{1}) - (0.03F_{1})^{2}] + 0.5[\sigma^{2}(F_{2}) - (0.03F_{2})^{2}] + (0.03F_{av})^{2}$$

where the terms in brackets were set equal to zero if they were negative. If the expression  $\sigma^2(F_{av}) = 0.5(F_1 - F_2)^2$  gave a larger number, it was used instead. In no case was any reflection of the combined set allowed to have a standard deviation less than 3% of itself (corresponding to 6% of I). A plot of the resulting  $\sigma(F_{\rm o})$  vs.  $F_{\rm o}$  revealed that the great majority of points fall on or near a smooth curve. This curve starts near 10 electrons for small  $F_0$ , stays near 3 electrons for  $F_0 = 40-100$  electrons, and follows the 3% of  $F_o$  line thereafter. Forty-five per cent of the data have  $F_{\circ} \ge 70$  electrons, and, of these, only about 8% have standard deviations more than three electrons above the curve described, with the 002 reflection by far the worst at 441  $\pm$  66 electrons. These large deviations were, of course, caused by disagreements between the two data sets obtained from the two different crystals. All data were corrected for Lorentz and polarization effects, but no corrections beyond the averaging just described were made for absorption or extinction.

Computations were performed on an IBM 7044 and a CDC 6600. Zalkin's FORDAP and DISTAN programs (unpublished) were used for Fourier syntheses and distance and angle calculations. An unpublished Wilson-plot program by Maddox and Maddox gave normalized structure factor magnitudes<sup>11</sup> which were used in Long's sign-determining program<sup>12</sup> as described below. Our unpublished version of the Gantzel-Sparks-Trueblood leastsquares program minimizes the function  $\Sigma w(|kF_0| - |F_0|)^2/$  $\Sigma w | k F_o |^2$ , where  $F_o$  and  $F_o$  are the observed and calculated structure factors, k is the scale factor, and w is the weighting factor. Scattering factors<sup>13</sup> for Co<sup>3+</sup> and neutral Br were corrected for the real part of anomalous dispersion by -2.2 and -0.9 electrons, respectively. The imaginary part of anomalous dispersion was neglected. Scattering factors for neutral nitrogen, carbon, and boron were also taken from standard tables.<sup>13</sup> The anisotropic temperature factors used have the form:  $\exp(-0.25\Sigma_i\Sigma_jh_ih_jb_i)$  $b_j B_{ij}$ , i, j = 1, 2, 3, where  $b_i$  is the *i*th reciprocal cell length.

#### Results

The monoclinic unit cell, space group P2<sub>1</sub>/c,  $a = 19.893 \pm 0.010$  Å,  $b = 19.487 \pm 0.010$  Å,  $c = 15.058 \pm 0.010$  Å,  $\beta = 93.15 \pm 0.05^{\circ}$ , contains eight formula units of N(CH<sub>3</sub>)<sub>4</sub>[(B<sub>9</sub>C<sub>2</sub>H<sub>8</sub>Br<sub>3</sub>)<sub>2</sub>Co]. The calculated density of 1.967 g/cm<sup>3</sup> agrees with the value (1.98  $\pm 0.01$  g/cm<sup>3</sup>) found by flotation in a mixture of bromoform and ethylene dichloride. These measurements apply to room temperature (~23°) and the error figures given are estimates. The observed extinctions correspond to space group P2<sub>1</sub>/c. All four of the twofold sets of special positions are occupied by cobalt atoms and all other atoms are in general fourfold positions  $\pm (x, y, z; x, \frac{1}{2} - y, \frac{1}{2} + z)$ .

#### **Determination of Structure**

Attempts to interpret the Patterson function calculated from the second (complete) data set failed because of an error in communication which resulted in the mis-

(13) "International Tables for X-Ray Crystallography," Vol. III, The Kynoch Press, Birmingham, England, 1962, pp 204-207, 214.

<sup>(10)</sup> It may be argued that  $F^2$  should be the quantity averaged, but the "error" incurred is only 5.4% even if  $F_1 = 2F_2$   $(I_1 = 4I_2)$ .

<sup>(11)</sup> J. Karle and I. L. Karle, Acta Cryst., 21, 849 (1966).

<sup>(12)</sup> R. E. Long, Ph.D. Thesis, University of California, Los Angeles, Calif., 1965.

	Final Positional and Thermal Parameters <sup>a</sup> in $N(CH_3)_4[(B_3C_2H_8Br_3)_2C_0]$								
Atoms	x	y	z	$B_{11}$	$B_{22}$	$B_{33}$	$\mathcal{B}_{12}$	B13	$B_{23}$
1 Co	0	0	0	3.1(2)	2.1(2)	3.0(2)	0.7(2)	0.1(2)	0.0(2)
$1 \operatorname{Br}(5)$	$0.0939(1)^{b}$	0.0812(1)	-0.1619(2)	6.1(1)	5.8(2)	5.0(2)	-0.1(1)	0.9(1)	0.6(1)
$1 \operatorname{Br}(9)$	0,1640(2)	0.1975(2)	0.0155(2)	7.9(2)	6.9(2)	8.3(2)	-2.5(1)	-1.2(2)	0.2(2)
1 Br (10)	-0.0010(1)	0.2408(1)	-0.1278(2)	7.6(2)	3.4(1)	8.4(2)	-0.1(1)	-1.5(2)	2.2(1)
2 Co	0.5	0.5	0	2.2(2)	2.2(2)	2.2(2)	-0.1(2)	0.4(2)	-0.2(2)
$2 \operatorname{Br}(5)$	0.3829(1)	0.4459(1)	0.1533(2)	4.9(1)	4.7(1)	5.4(2)	-0.8(1)	2.5(1)	0.4(1)
$2 \operatorname{Br}(9)$	0.3712(2)	0.2769(2)	0.0308(2)	12.7(2)	8.2(2)	6.5(2)	-7.7(1)	1.4(2)	-1.0(2)
2 Br(10)	0.5025(1)	0.3069(1)	0.2262(2)	8.3(2)	5.0(1)	4.8(2)	-1.0(1)	0, 2(1)	2.0(1)
3 Co	0	0.5	0	2.1(2)	2.1(2)	3.1(3)	0.3(2)	0.3(2)	0.0(2)
$3 \operatorname{Br}(5)$	0.0684(1)	0.6171(1)	-0.1475(2)	5.1(1)	5.9(1)	8.4(2)	0.1(1)	1.8(1)	4.2(1)
$3 \operatorname{Br}(9)$	0.1905(1)	0.4844(2)	-0.2110(2)	6.2(1)	7.8(2)	6.9(2)	-1.3(1)	3.5(1)	-2.1(1)
$3 \operatorname{Br}(10)$	0.2332(1)	0.6034(2)	-0.0147(2)	4.3(1)	8.4(2)	8.6(2)	-3.1(1)	1.3(1)	-2.1(2)
4 Co	0.5	0	0	2.6(2)	2.5(2)	2.9(3)	0.9(2)	0.4(2)	0.2(2)
$4 \operatorname{Br}(5)$	0.4304(1)	-0.1082(1)	-0.1688(2)	5.8(1)	4.2(1)	3.3(1)	-0.5(1)	0.4(1)	-0.6(1)
$4 \operatorname{Br}(9)$	0.2666(1)	-0.0179(2)	-0.1510(2)	4.8(1)	7.5(2)	6.9(2)	0.1(1)	-2.0(1)	1.8(2)
$4 \operatorname{Br}(10)$	0.3133(1)	-0.1774(1)	-0.0066(2)	6.4(1)	4.7(1)	5.0(2)	-1.7(1)	0.6(1)	0.0(1)
Atoms	x	У	z	В	Atoms	x	У	z	В
1 C(2)	-0.060(1)	0.070(1)	0.057(1)	3.5(5)	3C(2)	0.068(1)	0.463(1)	0.088(2)	4,8(6)
1 C(3)	0.012(1)	0.050(1)	0.115(2)	4.7(6)	3 C(3)	0.053(1)	0.413(1)	0.004(1)	4.4(5)
1 B(4)	0.079(1)	0.058(1)	0.058(2)	3.8(6)	3 B(4)	0.056(1)	0.451(1)	-0.099(2)	3.9(6)
1 B(5)	0.044(1)	0.091(1)	-0.055(2)	3.4(6)	3 B(5)	0.085(1)	0.540(1)	-0.067(2)	2.2(5)
1 B(6)	-0.040(1)	0.092(1)	-0.042(2)	3.1(6)	3 B(6)	0.090(1)	0.546(1)	0.052(2)	3.9(6)
1 B(7)	-0.034(2)	0.124(2)	0.141(2)	7.5(9)	3 B(7)	0.131(2)	0.398(2)	0.056(2)	7.0(9)
1 B(8)	0.060(1)	0.124(1)	0.136(2)	4.3(7)	3 B(8)	0.122(1)	0.399(2)	-0.053(2)	5.7(8)
1 B(9)	0.079(1)	0.150(2)	0.029(2)	6.2(8)	3 B(9)	0.147(1)	0.477(1)	-0.096(2)	3.3(6)
1 B(10)	0.005(1)	0.169(1)	-0.036(2)	3.8(6)	3 B(10)	0.164(1)	0.532(1)	-0.009(2)	3.5(6)
1 B(11)	-0.068(2)	0.151(2)	0.033(2)	7.4(9)	$3 \operatorname{B}(11)$	0.159(2)	0.483(2)	0.091(2)	6.2(8)
1 B(12)	0.011(2)	0.192(2)	0.073(2)	7.7(9)	3 B(12)	0.185(1)	0.445(2)	-0.007(2)	5.6(8)
2 C(2)	0.580(1)	0.435(1)	-0.001(1)	4.3(6)	4 C(2)	0.447(1)	-0.009(1)	0.113(1)	3.4(5)
2 C(3)	0.522(1)	0.422(1)	-0.082(1)	4.1(5)	4 C(3)	0.425(1)	0.060(1)	0.053(1)	3.7(5)
2 B(4)	0.443(1)	0.413(1)	-0.048(2)	3.9(6)	4 B(4)	0.407(1)	0.037(1)	-0.064(2)	3.9(6)
2 B(5)	0.455(1)	0.418(1)	0.073(2)	1.8(5)	$4 \operatorname{B}(5)$	0.416(1)	-0.055(1)	-0.063(2)	4.5(7)
2 B(6)	0.541(1)	0.436(1)	0.101(2)	2.9(6)	4 B(6)	0.445(1)	-0.085(1)	0.046(2)	3.7(6)
2 B(7)	0.578(2)	0.357(2)	-0.050(2)	6.7(8)	4 B(7)	0.372(2)	0.030(2)	0.128(2)	6.7(8)
2 B(8)	0.488(1)	0.342(2)	-0.079(2)	6.4(8)	$4 \operatorname{B}(8)$	0.346(1)	0.060(1)	0.018(2)	4.5(7)
2 B(9)	0.447(1)	0.340(2)	0.023(2)	5.7(8)	4 B(9)	0.336(1)	-0.017(1)	-0.052(2)	3.4(6)
2 B(10)	0.509(1)	0.352(2)	0.109(2)	5.4(7)	4 B(10)	0.357(1)	-0.088(1)	0.014(2)	2.9(6)
2 B(11)	0.589(1)	0.361(1)	0.064(2)	4.7(7)	4 B(11)	0.378(1)	-0.060(1)	0.124(2)	4.7(7)
2 B(12)	0.527(2)	0.301(2)	0.019(2)	6.3(8)	$4 \operatorname{B}(12)$	0.311(1)	-0.020(1)	0.060(2)	5.0(7)
1 N	0.773(1)	0.255(1)	0.265(1)	4.9(4)	2  N	0.269(1)	0.259(1)	0.272(1)	6.3(5)
$1 \operatorname{CH}_8(1)$	0.796(2)	0.214(2)	0.345(3)	14.8(13)	$2 \operatorname{CH}_{3}(1)$	0.237(2)	0.301(3)	0.196(3)	17.4(16)
$1  \text{CH}_{3}(2)$	0.701(2)	0.245(2)	0.250(2)	11.1(10)	$2  \text{CH}_{3}(2)$	0.240(3)	0.216(3)	0.337(5)	24.5(24)
$1  CH_3(3)$	0.812(1)	0.235(1)	0.187(2)	8.1(8)	$2 \operatorname{CH}_8(3)$	0,312(2)	0.209(2)	0.233(3)	17.3 (16)
$1  CH_3(4)$	0.791(2)	0.329(2)	0.300(3)	12.6(11)	$2  CH_3(4)$	0.301(2)	0.317(2)	0.328(3)	14.0(13)

TABLE I FINAL POSITIONAL AND THERMAL PARAMETERS<sup>a</sup> IN N(CH<sub>2</sub>)4[(B<sub>2</sub>C<sub>2</sub>H<sub>2</sub>BI<sub>2</sub>)<sub>2</sub>C<sub>0</sub>]

<sup>a</sup> The units are  $Å^2$ . <sup>b</sup> The numbers in parentheses are the standard deviations in the least significance digits as calculated from the final diagonal least-squares matrix. They are not significantly different from those obtained from the complete matrix (see text and footnote 15).

taken idea that there were four, rather than eight, molecules per unit cell. Normalized structure factor magnitudes,<sup>11</sup>  $E_h$ , were calculated and used in Long's signdetermining program<sup>12</sup> which iteratively applies the Sayre relation:  $sE_h \sim s\Sigma_k E_k E_{h-k}$ , where s is to be read "the sign of." After enough signs were worked out by hand to yield a good set of "starting reflections" for input,<sup>14</sup> a run of Long's program on 379 E's ( $\geq 1.5$ ) gave one set of signs which was better than any other as judged by the smaller number of passes necessary to reach it and by its high consistency index (0.82 as against the second-best 0.65). The consistency index for the signed E's is defined as:  $\Sigma_h \Sigma_k E_h E_k E_{h-k}/$ 

(14) The origin was defined by assigning positive phases to  $42\overline{3}$ ,  $71\overline{1}$ , and 874. All combinations of positive and negative phases were assigned to 441, 640, and  $78\overline{7}$  to generate the successive sets of signs.

 $\Sigma_h \Sigma_k [E_h E_k E_{h-k}]$ . A Fourier synthesis using the signed E's as coefficients showed 16 largest peaks ranging in relative size from 1.00 to 0.57 with a definite breakdown to the 17th at 0.28. Isotropic bromines at these 16 positions, 4 of which were the twofold special positions, quickly refined to R = 0.26 where  $R = \Sigma ||kF_o| |F_{\rm c}|/\Sigma |kF_{\rm o}|$ . A difference Fourier synthesis phased by these 16 atoms showed all 54 of the nonhydrogen light atoms. Their locations showed that the asymmetric unit contains 4 half-anions plus 2 tetramethylammonium ions and that the cobalt atoms are those in the 4 twofold sets of special positions. The refinement beyond this point was routine except that, because of the large number of parameters involved, it was done in a block-diagonal fashion. The heavy atoms (4 anisotropic cobalts fixed at the special positions plus 12 anisotropic bromines) were refined by a full matrix while the light atoms were held fixed, alternately with fullmatrix refinements of the 54 isotropic light atoms with the heavy atoms fixed. The 2 carbons in each of the 4 icosahedral fragments were identified by their low thermal parameters when refined as borons and the shorter distance between them. The partial data set from the first crystal was averaged in as described above when the refinement halted at R = 0.11 and it was found that the large disagreements were concentrated at low angle (where most of the partial data set had been obtained). Continued refinement using this composite data set changed the structure only slightly, but R fell to nearly its final value and the distribution of large disagreements became more uniform. Six cycles of diagonal-matrix least squares (all parameters) finished the refinement and gave the final R value of 0.087. The standard deviation of an observation of unit weight, defined as  $[\Sigma w(|kF_o| - |F_o|)^2/(u - v)]^{1/2}$ , where u is the number of data and v is the number of independent parameters refined, was 2.28. Standard deviations derived from the final diagonal matrix were typically 10% less than those obtained from one cycle of full-matrix refinement which included all parameters, and ranged from approximately 5% larger to approximately 25% smaller.<sup>15</sup> In the last cycle, no parameter shifted more than 4% of the standard deviation calculated for it, except for the tetramethylammonium ions, where a few parameters shifted by as much as 11% of their calculated standard deviations. The six largest peaks in a final difference Fourier (1.9, 1.8, 1.8, 1.3, 1.1, and 0.9  $e^{-/\text{Å}3}$  were all judged to be due to error in the data, since their positions (far from any atom) made no chemical sense. This result made a search for hydrogens unjustified. The final atomic coordinates and thermal parameters are listed in Table I and the values of  $|kF_o|$  and  $F_o$  are given in Table II.

#### Discussion

All four crystallographically unrelated anions were found to have the same atomic arrangement with corresponding dimensions equal to within the experimental accuracy. Figure 1 illustrates the structure of the Co- $(B_9C_2H_8Br_3)_2^-$  anion and the numbering system used in this study. The top and bottom halves of the anion are related by a crystallographic inversion center located at the cobalt. Interatomic distances are presented in Table III and averages thereof are presented in Table IV. The average B–B, B–C, C–C, B–Br, and Br–Br distances are in agreement with those found<sup>16–21</sup>



Figure 1.—Structure of  $(B_9C_2H_8Br_3)_2Co^-\ (hydrogens\ omitted).$ 

for similar compounds. The Br-B-B angles are all (with one exception) within  $5^{\circ}$  of their  $121^{\circ}$  average and show no significant deviations from this value which are systematic over the four anions. The Br(5)-B(5)-Co angles are slightly smaller  $(116-119^{\circ})$  as expected from the protrusion of the cobalt vertex of the icosahedron. This protrusion is also shown by the smaller angles (49.4° with a root-mean-square deviation of  $\pm 1.7^{\circ}$  about this value) at cobalt. The 120 B-B-B angles necessarily average to exactly 60° and have a root-mean-square deviation of 2°. The 16 B-C-B and 32 C-B-B angles average 63 and 58.5° while the 4 C-B-C and 8 B-C-C angles average 57 and  $61.5^{\circ}$ . All four of these sets of angles show  $2.5^{\circ}$  rootmean-square deviations from these averages. The root-mean-square deviations are believed to be better measurements of the accuracy of these angles than the  $\sim 1.6^{\circ}$  values derived from the standard deviations of coordinates. The temperature factors in the dicarbollyl portion of the anions seem to indicate some rigidbody motion, but a rigid-body analysis was not done because of the excessive effort it would require.

The packing of the approximately dumbbell-shaped anions and the tetramethylammonium ions is very similar to that of  $\text{KHF}_2^{22}$  as indicated in Figure 2. The higher symmetry (tetragonal, I4/mcm) of the  $\text{KHF}_2$ structure<sup>22</sup> is broken down to its P2<sub>1</sub>/c subgroup (on the doubled cell) by the lack of cylindrical symmetry of the anion and the up and down (in z) distribution of the bromine substituents. If we consider only the arrangement of plus and minus charges, this is approximately a CsCl-type structure.

The pentagonal rings adjacent to cobalt are all planar to within the accuracy of this determination but are tilted or slid over in such a way as to decrease the cobalt-carbon distance and increase the cobalt-boron distances (Table IV). The tilt and slide descriptions are

<sup>(15)</sup> Late in the refinement, one cycle of full-matrix least squares was run on all 361 parameters (12 fixed) and 3002 data to be sure that no errors were caused by the block-diagonal procedure. This one cycle required 74K storage and 43 min on a CDC 6600. These figures may be compared with the requirements for the final diagonal-matrix refinements of 28K storage and 2.24 min/cycle.

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<sup>(20)</sup> J. A. Potenza and W. N. Lipscomb, *ibid.*, 5, 1471, 1478, 1483 (1966), and references therein.

<sup>(21)</sup> J. A. Potenza and W. N. Lipscomb, ibid., 3, 1673 (1964).

<sup>(22)</sup> R. W. G. Wyckoff, "Crystal Structures," Vol. 2, Interscience Publishers, New York, N. Y., 1964, p 277 ff.

H.X= 0, C L FOS FCA 2 441 921	1 116-121 2 110 110 3 42 -49	-1 143 147 6 275-237 1 c4 58	2 45 -37 3 21 -9 4 47 -52	H.K# 2, 5 L FOB FCA	H,K= 2+ 12 L F08 FLA -7 32 +42	1 187 195 2 251-269 3 90 -84	0 114 115 1 114 106 2 164-165	-4 150-125 -3 116 -95 -2 69 -78	1 88 -83 2 138-146 3 64 67	8 56 62 9 21 1 10 30 41	-3 105-103 -2 42 34 -1 165 163	3 106 115 4 74 86 5 14 -0	-2 45 45 -1 10 -12 0 66 71	L FOE FCA -9 93 -77 -8 154 98
4 224 203 6 578 015 8 248 254 10 32 -31	4 113 110 5 147 134 0 53 59 7 03 -07	2 161 205 3 109 115 4 112 -96 5 155 151	5 55 -67 6 59 57 7 65 64	-1: 67 63 -10 52 -60 -9 42 40 -8 54 -59 -7 14 -21	-6 33 26 -5 122-127 -4 60 59 -3 17 13	4 96 89 5 31 45 6 79 78 7 36 ~31 8 105-119	3 48 40 4 60 -49 5 53 48 6 155 159 7 56 -50	-1 79 70 0 270 224 1 532-528 2 150-165 3 48 67	4 12 -6 5 74 -81 6 11 7 7 62 -59	M,K# 5+ 5 L FOB FCA -10 0 1 -9 49 -40	0 20 -34 1 116-120 2 121-113 3 62 -56 4 21 -17	6 63 -72 7 124 129 6 23 -16 9 61 -64 16 79 63	1 14 -30 H.K= 7, 0 L FUB FCA -10 37 -34	-7 45 -46 -6 17 -13 -5 0 -4 -4 99 160 -3 145-142
H,K= 0, 1 1 FE8 FLA 1 7 -4 2 128-155	F,K= 0, 13 L FGo FCA L δ 10 2 111 111	7 61 77 6 93 96 9 113 112 10 16 27	L FOB FCA -6 155 155 -5 40 -34 -4 47 53	-6 210 222 -5 4L -43 -4 127 156 -3 56 -5d	-1 101-109 0 94 78 1 19 17 2 165 159	9 28 20 10 22 -30 11 20 -2	H,K= 3, 12 L FD5 FC4 -7 39 -48	4 67 -57 5 37 -37 6 1J3 104 7 109-105	H+K= 4, 12 L FOd FCA -5 36 44 -5 50 -50	-6 132 134 -7 3 6 -6 40 63 -5 12 -2	5 15 12 6 100-104 H.K= 5, 13	Hyk= 6, 0 L FDD FCA -10 55 42	-8 41 21 -6 65 -61 -4 40 -45 -2 94 80	-2 142 144 -1 04 -04 0 35 -15 1 36 -10
3 65 -92 4 47 -61 5 34 -35 6 217-235	3 139-140 4 20 21 5 27 -23 6 14 2	11 4 -12 Hyke 1, o L FOD FGA	-3 114-121 -2 32 41 -1 120 117 0 147 147	-2 34 -53 -1 225-268 6 114 99 1 257-244	3 108-105 4 44 40 5 18 12 5 137 98	H,K= 3, 5 L FCb FCA -11 101 -99 -10 127 115	-6 C 22 -5 12 2 -4 61 88 -3 76 83	8 174 181 9 48 -47 10 26 -14	-4 51 44 -3 180 181 -2 148 132 -1 16 5	-4 11 20 -3 13 22 -2 74 74 -1 72 -62	L FCb FCA -5 46 -39 -4 26 -29 -3 29 39	-9 102-102 -6 54 59 -7 85 92 -0 195 191	0 130-132 2 78 65 4 185-175 6 104 68	2 24 27 3 53 -52 4 14 16 5 43 -53
7 110 136 5 148-156 5 159-164 10 34 -91	<pre>&gt;&gt; K= 0, 14 L FGo FCA 0 44 - 54</pre>	-10 43 -40 -5 62 60 -6 70 -64 -7 151 152	1 151 151 2 147 151 3 27 -35 4 39 44	2 266 289 3 101-105 4 35 -12 5 328-366	7 76 -68 Hix# 2, 13 L FOB FCA	-9 80 80 -8 52 48 -7 41 41 -6 26 -27	-2 76 75 -1 46 52 0 63 57 1 72 -67	H,K= 4, 5 L FOB FCA =10 112=112 -9 107=104	C 226 212 1 95 90 2 210 199 3 140 138	0 95 78 1 11 16 2 11 3 3 51 49	-2 126-139 -1 66 -72 0 14 -14 1 44 -38	-5 95 96 -4 384 384 -3 76 -74 -2 127 95	8 68 762 10 49 35 H,K= 7, 1	8 60 61 7 57 78 8 0 14
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5 52 -62 6 175 160 7 13 -16 8 79 -62	F,K= 1, 0 L FOD FUA -10 ∠7 36	3 62 -91 4 60 77 5 57 76 6 6 0	0 29 -22 1 149 171 2 e3 -93 3 32 31	-10 82 90 -9 29 -24 -8 271 272 -7 136-130	3 19 -15 4 50 62 5 66 -72 6 138-136	4 234 220 5 215 223 6 186-209 7 137-139	-5 27 -31 -4 64 -59 -3 47 -48 -2 115-121	1 417 389 2 41 53 3 13 -27 4 24 6	-2 66 67 -1 44 -52 0 61 60 1 104 156	L FOB FCA -10 12 -4 -9 48 -46 -8 97 -66	0 30 -29 1 30 26 2 6 -14	8 56 6J 9 30 24 HiK= 6, 7	-2 142-155 -1 486 466 0 116 110 1 42 -56	-1 71 -70 6 107 103 1 80 79 2 144-155
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L FOB FCA 1 55 -/3 2 256-308 3 97-115	2 216 238 4 451-461 6 35 40 8 71 71	H,K= 1, 7 L FOL FLA -10 80 -75 -9 0 -7	-1 45 50 0 55 -59 1 24 23 2 34 -26	-1 375-351 G 188-164 I E1 73 2 34 -23	-2 103 114 -1 19 -16 0 76 75 1 59 63	L FOD FCA -10 79 -67 -9 52 -36 -8 0 5	4 24 6 5 54 -55 H,K= 3, 14	10 33 20 ≻,K= 4, 6 € FOB FCA	H,K= 4, 14 - FUB FCA -4 69 61 -3 75 -69	-2 69 -70 -1 17 8 0 240-197 1 92 82	-4 331 336 -2 373 327 0 64 56 2 50 35	-5 32 -21 -4 16 -0 -3 24 -15 -2 88 94	7 17 25 6 30 -16 9 54 -59 10 98 99	ε 22 -22 H,R= 7, -2 L FLB FLA
4 159 143 5 1e2-151 6 235 260 7 53 -51	10 20 -41 ⊢,K≈ 1, 1 L FUO FLA	-8 49 49 -7 20 -24 -8 82 -66 -5 99 -19	H,K* 2, 5 L FUB FCA -10 /9 -92	3 270-263 4 302 298 5 55 -64 6 49 -44	2 197 210 3 10 10 4 121 136	-7 61 -55 -6 24 26 -5 124-112 -4 154-177	L FOB FCA -4 27 3 -3 138 150 -2 0 16	-10 55 -37 -9 0 8 -8 31 -20 -7 270 263	-2 16 20 -1 13 -16 0 39 -20 1 35 -31	2 190-193 3 155 125 4 32 21 5 193-194	4 486 496 6 22 -5 8 102 96 10 253 247	-1 41 51 0 251-253 1 171-173 2 101-155	H.K.= 7, 2 1 FDB FLA =10 55 -43	-8 28 -31 -7 48 79 -6 45 95 -5 143 163
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H.K= 0, 4 L FOB FCA 0 269 265 1 70 -71	-0 10 -19 -5 214 234 -4 121 126 -3 57 -75	1 300-322 2 10 3 3 175 180 4 07 -72	2 433 414 4 339-32c 5 25 -71 8 116 122	H,X= 2, 7 U FJB FCA =10 10 =0 -9 57 02	1 36 -32 2 123 118 mak# 3, 0	2 22 -25 3 32 45 4 235 221 5 133-134	4 63 69 H,K= 3, 15 L FOL FGA	-1 204 201 0 2/8 247 1 146 139 2 25 6	-10 16 9 -8 291-3.3 -8 99 89 -4 51 57	H,K= 5, 7 1 FO6 FCA -10 0 -10	-9 48 57 -6 90 -95 -7 125 141 -6 78 91	5 107-105 5 120-110 F.K. 0, 0	-4 132 119 -3 119 127 -2 27 -22 -1 309 373	1 (36 137 2 196 197 3 61 -55 4 173 125
2 20 - 23 3 132 147 4 115 143 5 139-150 6 52 - 54	-2 179-225 -1 71 -67 0 6 -7 1 66 75 2 181 201	5 51 -53 6 23 in 7 85 -07 8 33 -27 9 25 15	H,K= 2, 1 L FDB FJA -11 45 0	-7 143-151 -6 72 67 -5 36 20 -4 72 -67	-10 62 65 -8 129 115 -6 216 237 -4 29 -73	7 94 105 8 116-121 9 77 65 10 72 67	0 77 -69 1 99 -91 H,K= 4, 0	4 153 144 5 122 125 6 153 167 7 46 37	0 138 130 2 420 426 4 174-144 5 52 52	-7 191-129 -6 26 19 -7 87 95 -6 248-258 -5 48 -26	-4 50 -57 -3 235 225 -2 227-230 -1 195 183	-9 97 -91 -8 57 -45 -7 81 -85 -6 21 16	0 75 -52 1 167-150 2 93 -65 3 87 98 4 20 4	5 36 59 6 107-111 7 99 103
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5 43 -37 6 23 -4 7 16 -3 8 156 157	4 164 198 5 195-208 6 53 53 7 97 97	L FOE FCA -9 95 102 -6 15 -9 -7 27 -37	-9 47 4/ -6 36 40 -7 208-215 -6 106 103	0 105 83 1 107 156 2 186 149 3 04 -68	6 48 -58 7 39 39 8 116-115 9 73 64	-10 24 -6 -9 74 73 -8 21 -21 -7 54 43	1 66 64 2 320 348 3 230-210 4 207 204	h,K= 4, 6 L FOB FCA -9 115-112 -8 181 168 -7 51 59	11 27 -46 H <sub>2</sub> K= 5, 2 L F05 FLA =11 23 =19	1 145 137 2 17 17 3 16 18 4 74 64 5 79 -79	2 21 2 3 182-169 4 299 279 5 11 14 6 111 112	2 221 225 3 66 58 4 26 -20 5 65 69	8 19 -3. 9 76 -81 10 39 -4.	2 40 -32 3 81 77 4 28 43 5 90 55
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1 20 14 2 71 -79 3 42 43 4 60 -62 5 18 17	H:K= 1, 3 L FOB FCA -11 143 139 -10 168 178 -9 167-160	-1 23 -9 U 76 69 1 97 -81 2 15 -25 3 195 220	0 140 91 1 217-221 2 412 403 3 90 91 4 57 54	9 50 -46 H+K= 2, 9 L FE8 FCA -9 71 73	-11 72 59 -10 51 -43 -9 0 -14 -0 31 -41 -7 96 -94	-1 182-167 0 53 56 1 190-171 2 20 10 3 711-225	10 75 70 11 23 19 F,K= 4, 2 L FOL FC4	-2 224 208 -1 130 128 0 302 296 1 34 -41 2 130 125	-5 114 136 -5 135-121 -4 363 352 -3 137-140 -2 274-262	H,K= 5, 9 L FO8 FC4 -9 6 -13 -8 33 -47	F+K= 6, 3 £ FOB FCA -10 31 -10 -9 81 -75	L FOL FCA -6 53 52 -7 123-124 -0 16 -9 -5 193 197	-7 0 -11 -6 93 -66 -5 157-167 -4 140-125 -3 141 141	-4 96 -92 -3 27 .4 -2 23 55 -1 175 175 6 97 1.5
6 43 52 7 33 33 8 17 18 9 19 6	-8 49 -42 -7 145 155 -6 64 71 -5 62 93	4 10 5 5 23 -31 6 19 -6 7 51 -44	5 105-196 6 105 105 7 261-296 8 55 57	-6 96 96 -7 105 106 -6 192 197 -5 106 103	-6 52 -66 -5 143 147 -4 103-119 -3 55 60	4 79 -77 5 96 -89 6 111-116 7 54 -56	-11 47 -39 -10 252 197 -9 167 184 -8 26 22	3 111-114 4 30 25 5 07 50 6 142 146	-1 47 53 5 27 25 1 317-352 2 50 -49	-7 159 161 -6 72 79 -5 123 125 -4 48 -49	-0 22 -25 -7 145-145 -0 140-142 -5 31 -33	-4 309 297 -3 93 97 -2 100 94 -1 36 -22	-2 39 36 -1 66 95 0 139 137 1 11 21	1 41 -41 2 38 -34 3 30 27 4 16 5
10 99 -95 H+K= 0+ 5 L FCB FCA	-4 279 310 -3 207-237 -2 209 253 -1 237-231 	6 17 5 9 52 60 F,K= 1, 10	9 141 139 10 123 120 11 37 -22 8.68 2. 4	-4 90 65 -3 44 44 -2 56 33 -1 204 200 6 151 157	-2 33 41 -1 60 47 0 116-105 1 45 41 2 164-167	8 93 85 9 89 -64 H,K= 3, 9 1 ED6 EC4	-7 33 18 -6 317 348 -5 101-101 -4 206 268 -3 565 572	7 63 65 8 123 116 9 127-117	3 42 38 4 65 57 5 109 106 6 139-165 7 51 -56	-3 65 -71 -2 237-239 -1 20 -4 0 17 16 1 89 99	-4 123 161 -3 125-129 -2 99 99 -1 195-195 0 14 3	1 40 43 2 195 190 3 151 162 4 271 264	2 54 55 3 14 -5 4 135-135 5 257 269 6 342 150	H+K= 7, 13 L FCE FLA -3 46 57 -2 113 131
1 143-155 2 43 -47 3 39 -36 4 115-116	1 219-268 2 172 207 3 11 -20 4 17 -20	-9 100 -92 -8 81 -79 -7 22 19 -6 74 79	L FDB FCA -11 62 -45 -16 45 -37 -9 92 43	1 45 47 2 146 149 3 12 27 4 19 -27	3 199 232 4 124 109 5 8 -3 6 69 92	-9 23 14 -8 22 -33 -7 82 -85 -6 144 151	-2 399 394 -1 105 94 0 107 -74 1 22 12	L FD8 FC4 -9 35 20 -8 9 -4 -7 27 34	6 74 72 9 30 -23 15 11 14	2 56 60 3 157-154 4 170-121 5 48 -50	1 261-243 2 240 250 3 9 9 4 90 80	5 35 -50 6 25 -20 7 16 6	7 116-133 8 139 128 9 21 -9	-1 58 -52 5 33 -35 1 162 -56 2 37 54
5 5 - 17 6 68 70 7 80 - 84 8 64 75 9 79 - 84	5 143-152 5 87 104 7 165 167 8 112 124 9 13 -14	-5 88 92 -4 96 105 -3 120-133 -2 30 -45 -1 55 -54	-8 130 121 -7 166-179 -6 143-162 -5 117 127 -4 62 -57	5 56 60 6 72 64 7 42 -33 8 23 18 9 18 21	6 16 -13 9 12 6 16 85 -66 11 10 18	-5 159-175 -4 54 -51 -3 137-141 -2 46 35 -1 33 -19	2 429 415 3 362 394 4 265 253 5 231 244 5 80 70	-5 167-161 -4 29 -30 -3 31 31 -2 232-226	L FDS FLA -11 64 63 -10 71 -69 -9 45 -46	7 116 115 6 54 c0 M+K= 5+ 10	5 111-114 6 135 142 7 126-138 5 42 49 9 2617	-7 101 99 -5 57 62 -5 47 43	-10 74 -73 -9 67 65 -8 59 -54	7 40 44 M,K= 6, 0 L ftt Fuk -16 11 10
10 33 18 H,K# 0, 9 L FDB FGA	10 152-145 11 5 2 H,K= 1, 4	0 51 -42 1 72 87 2 175 185 3 114-124	-3 47 -44 -2 168 164 -1 69 62 0 172-176	H,K# 2, 10 L FO5 FUA -8 78 76	H,K= 3, 3 L FOB FCA -11 8 1 -10 53 50	0 59 55 1 128-119 2 18 21 3 174-170	7 22 -17 8 113 120 9 178 183 10 39 39	-1 74 81 0 95 89 1 38 -40 2 39 -40 3 28 -27	-5 70 64 -7 /1 68 -6 37 38 -5 50 -55 -4 305-278	L F08 FLA -8 143-132 -7 41 44 -6 73 -76 -5 12 11	10 10 -12 H,K= 0, 4 L FOS FCA =10 116 103	-4 123 130 -3 239 233 -2 19 27 -1 94 87	-7 63 -74 -6 56 50 -5 100 102 -4 100-113	-8 265 237 -6 32 48 -4 55 11 -2 146 131 0 125 75
2 46 -33 3 259-269 4 15 6 5 124-131	-11 23 -24 -13 77 66 -9 76 -73 -5 115 111	53 -57 5 45 42 7 95-103 5 35 32	2 114-127 3 209 210 4 43 43 5 175 183	-6 56 55 -5 130 134 -4 112 110 -3 69 -74	-9 15 -11 -6 42 -51 -7 9 -1 -6 105-137	5 65 67 6 76 -83 7 6 13 8 52 51	H <sub>1</sub> K= 4, 3 L FOB FCA =11 20 12	4 137-150 5 47 47 6 23 18 7 81 -85	-3 516 282 -2 125 133 -1 115-116 0 222 2J5	-4 103 103 -3 85 -78 -2 9 7 -1 39 -44	-9 31 38 -8 207 204 -7 53 46 -6 211 217	1 62 -65 2 126 127 3 196 190 4 125-123	-2 13 -34 -1 176 173 0 64 61 1 67 31	2 51 35 4 25 12 6 12 3 8 132 126
o 135 125 7 42 42 8 149-142 9 90 -67	-7 123-130 -6 61 -47 -5 175-173 -4 51 66 -3 119-115	H,K= 1, 11 L FUE FGA -6 55 53 -7 51 -59	6 106 121 7 27 -34 8 55 50 9 243 249 10 30 25	-2 25 -6 -1 68 -73 0 31 18 1 74 63 2 142-135	-5 14 -6 -4 119-123 -3 18 35 -2 178 194 -1 15 8	9 37 -48 H.K= 3, 10 L FOB FCA -8 169-157	-10 57 40 -9 73 72 -8 66 70 -7 72 72 -6 120-124	8 111 -99 +,K= 4, 10 L FOS FCA -8 33 46	1 55 53 2 320-313 3 338 315 4 8 28 5 88 -94	0 169-139 1 15 2 2 114 117 3 41 -43 4 81 77	-5 47 -32 -4 167 178 -3 136-135 -2 169 190 -1 232 135	5 77 77 5 34 -34 HyK= 5, 12	2 12 U 3 40 41 4 24 -17 5 19 -22 0 18 -14	H <sub>1</sub> K= 6 <sub>1</sub> 1 U FC6 FCA -10 U 9 -0 47 60
H,K= 0, 10 L FCB FCA C 245 268 L 123-L39	-2 24 -17 -1 161-176 G 9 4 1 115 111	-6 36 -33 -5 30 24 -4 91 -84 -3 7 -1	11 42 41 H.K= 2, 4 E FOB FCA	3 23 12 4 156 168 5 66 -71 6 80 62	0 55 -64 1 18 21 2 237-246 3 55 -54	-7 0 -11 -6 16 4 -5 32 34 -4 129 123	-5 226 216 -4 14 26 -3 51 -47 -2 15 18	-7 30 -31 -6 162 158 -5 15 16 -4 117 103	6 199 219 7 38 47 8 79 -70 9 53 53	5 26 9 6 41 37 7 59 42	0 545 494 1 104-104 2 56 68 3 6 14	-0 21 24 -5 25 11 -4 20 4 -3 85 80	7 50 -54 6 21 -31 9 40 47	-0 127-123 -7 79 -77 -6 55 113 -5 10 -10
2 84 100 3 143 141 4 279 290 5 112 118 6 226 222	2 65 -03 3 258-268 4 161-105 5 32 25 6 66 -64	-2 35 60 -1 45 -45 U 57 45 1 67 -70 2 78-125	-11 97 91 -10 170 170 -9 260 254 -8 227 215 -7 46 -61	7 33 -36 6 63 -73 H.K= 2, 11 L FOB FCA	4 159 165 5 46 42 6 49 39 7 51 -54 8 38 -28	-3 95 94 -2 49 -51 -1 103 -97 0 247-241 1 119 121	C 110-108 1 341 318 2 247-257 3 169-163	-2 91 79 -2 94 -83 C 163 171 1 87 -80	H,X= 5, 4 L FOE FLA -11 6 17	L FDB FLA -7 68 -65 -6 82 75 -5 74 69	4 38 10 5 43 41 6 250 249 7 95 96 8 26 28	-2 69 67 -1 203-203 0 108 101 1 106-108 2 86 65	H,K= 7, 5 L F03 FLA -9 47 44 -8 57 53 -7 133-135	-4 76 -75 -3 141-132 -2 203-174 -1 165-167 0 165-160
7 0 -18 8 63 64 3 60 65	7 49 53 a 133-135 9 0 -25 10 63 61 11 5 - 5	3 30 30 4 143-154 5 66 -87 6 199 202 7 101-100	-6 43 -47 -5 220 229 -4 231 237 -3 362 429	-8 23 -2 -7 79 -67 -6 21 -17 -5 26 28	9 80 -74 10 34 -34 11 22 20	2 152 163 3 50 52 4 115-120 5 76 74 6 35 - 21	4 132-109 5 45 -61 6 96-102 7 37 41 8 102-120	2 76 77 3 13 8 4 198 195 5 148-152 6 55	-10 19 -21 -9 44 -41 -8 68 65 -7 39 42 -6 26 20	-4 109 106 -3 13 -1 -2 39 -33 -1 96-106 0 222 223	9 U 14 10 17 27 H,K# 6, 5 U FDB FCA	3 5t -62 4 32 -10 5 65 -71 8,8= 6, 13	-6 52 54 -5 110 96 -4 64 60 -3 21 -10 -2 127 135	1 14 -13 2 13 21 3 54 453 4 115-134 5 94-154
L FDB FCA 1 145 L67 2 193-215 3 9 22	h 32 - 35 h K = 1, 5 L FOD FCA -11 52 - 47	H,K# 1, 12 L FUE FCA	-2 570 365 -1 151 154 C 155-147 L 64 75 2 408 397		L FOB FCA -11 59 47 -10 132-131 -9 51 80	7 11 15 8 15 30 H <sub>7</sub> K= 3, 11	9 5 12 10 125-109 11 62 -51	7 54 -65 8 69 65 H,X= 4, 11	-5 43 -62 -4 158-55 -3 64 55 -2 47 49	1 46 38 2 68 66 3 61 61 4 97-103	-10 24 22 -9 166-163 -8 36 39 -7 6 7	L FUE FCA -4 14 3 -3 67 -75 -2 39 -59	-1 137-131 0 36 20 1 77 -76 2 111 117	6 114-172 7 23 -1 8 5 0 9 63 70
4 90 93 5 39 44 6 29 31 7 131 132 8 115-11	-10 41 -48 -9 98 -63 -8 135-128 -7 13 13	-7 147-147 -0 11 19 -5 77 80 -4 23 -0	3 441 467 4 161 171 5 43 -47 6 152 156 7 7 1	1 20 15 2 03 -73 3 55 53 4 92 92 5 53 -5	-8 154 154 -7 78 86 -6 80 -62 -5 13 1 -4 385-384	L FD8 FCA -8 47 -56 -7 9 -12 -6 26 22 -5 112 12	H,K= 4, 4 L FOE FCA -11 112 -99 -10 92 82 -9 91 81	L FDB FCA -7 93 -81 -6 15 -21 -5 87 -82 -4 58 -5:	-1 00 -65 0 204 229 1 15 -26 2 107-100 3 51 -90	> 18 -8 6 165 161 7 42 -42	-6 40 -37 -5 13 -5 -4 63 47 -3 58 -46 -2 31 -39	-1 05 -64 0 10 1 1 40 -61 2 55 50 3 15 -7	3 94 38 4 13 -3 5 157-160 6 151 198 7 79 -73	
+,x= 0, 12 L FOB FCA C 132 127	-0 18 -6 -5 140 140 -4 30 37 -3 151-149 -2 265-319	-3 144-192 -2 122-128 -1 17 -24 0 74 65 1 0 10	8 166 170 9 131 125 10 186 179 11 25 -14	6 6 15 7 20 -12 8 72 74	-3 232 252 -2 461 451 -1 90 -79 0 100 89	-4 62 -71 -3 82 79 -2 63 -62 -1 65 49	-6 63 56 -7 111-113 -6 165 158 -5 321-343	-3 10 6 -2 40 39 -1 221-212 0 53 53	4 15 -5 5 41 29 6 13 17 7 104 -97	L FOB FCA -6 84 -60 -5 29 -23 -4 21 -9	-1 13 b 0 221-187 1 59 60 2 79 60	4 43 -50 F,K= 0, 14 L FUB FCA	8 30 -21 9 10 5 H,K= 7, 7	

TABLE II

# Observed and Calculated Structure Factors for $N(CH_8)_4[(B_9C_2H_8Br_8)_2Co]$

н,

H+K= 8+ Z	2 119-111	3 96 -93	1 102 94	-4 70 55	2 73 67	-5 99 102	-4 205 201	2 62 66	6 218 218	0 65 -69	-7 39 -34	5.K= 13. 0	2 44 41	
L FO8 FCA	3 170-169	4 70 65	2 81 83	-3 177 165	3 38 - 39	-4 154 152	-3 214 218	3 26 27	1 33 37	1 18 12	-6 36 -30	L FOS FCA	3 26 15	h,K= 14, 4
-10 3 27	4 42 37	5 42 - 37	3 190-191	-2 142-147	4 67 51	-3 65 -61	~2 227 222		2 74 05	2 60 -54	-5 45 43	-0 59 65	4 19 13	L FOS FCA
-9 118-109	5 133-130	6 15 1	4 70 -75	-1 114-138	5 14 2	-2 88 88	-1 102-101	H,K= 11, 0	3 160 -69	3 26 27	~4 102 102	-4 49 -35		-4 41 48
-8 146 155	6 18 35	1 03 03	5 4 9	0 105 106	6 70 -72	-1 43 -49	0 122 130	E FDB FCA	4 76 -89	4 25 36	-3 53 50	-2 149 165	H + K = 13, 0	-3 19 6
-6 247 265	8 101-100	H.K= 6, 10	7 16 -20	2 125 126	H.K= 9, 10	1 142 136	2 87 79	-6 48 -50	5 70 /1	H.K= 11. 10	-1 99 98	2 207 213	-5 31 39	~1 33 37
-5 25 -25	9 112-121	L FD6 FCA	8 12 22	3 111 111	L FOB FCA	2 62 63	3 216 225	-4 291-290	7 36 - 39	L FCB FCA	0 51 77	4 14 -5	-4 38 36	0 22 20
-4 27 -24		-7 11 -31	9 12 ~5	4 51 -55	~6 93 104	3 118-129	4 293 296	-2 55 59		-3 13 -12	1 26 - 30	o 53 59	-3 32 -23	1 126-129
-3 104-113	H,K= 0, D	-6 42 30	H.K. 9. 2	5 38 - 39	-5 31 -19	4 140 142	5 10 12	0 119 139	H,K= 11, 5	-2 30 -24	2 188 161	5. K. 13. I	-2 75 69	2 15 9
-1 35 - 34	-9 126-114	-4 243 240	L FUB FCA	7 36 -36	-3 115 117	6 75 79	7 50 -52	4 117-116	-7 65 -/0	0 53 - 55	4 55 58	L FOD FCA	0 117-113	5 10 -18
0 34 -19	-8 122-11Z	-3 39 39	-9 74 -74	8 97 100	∽2 b7 88	7 39 42		6 139 153	-6 62 -75	1 30 - 38	5 71 73	-7 122-134	1 40 -46	H,K= 14, 5
1 220 206	-7 60 -71	-2 141 140	-8 103 -96		-1 65 58	8 58 - 55	H,K= 10, 7	8 93 -91	-5 137-142	2 16 -14	o 53 56	-6 17 -3	2 69 74	L FCb FLA
3 172-166	-6 /9 /6	0 30 -12	-6 28 = 30	H K= 9, 6	0 24 14	H.K- 10. 3	L FUS FUA	N.K. 11. 1	-4 10 9	3 5 - 10	W-F- 12. 5		3 21 3 6 53 65	-4 24 11
4 110 117	-4 85 79	1 19 11	~5 179-176	-8 129-130	2 23 16	L FOB FCA	-6 76 -79	L FOB FLA	-2 29 -18	H,K= 12, 0	L FOU FCA	-3 45 40	4 37 03	-2 72 -62
5 87 72	-3 82 -88	2 178 167	-4 64 50	-7 31 20	3 54 47	-9 59 54	-5 31 20	-8 52 42	-1 24 -33	L FOB FCA	-7 95 97	-2 36 46	H:K= 13, 7	-1 26 -15
6 219 232	-2 124-113	3 0 -10	-3 148-156	-6 60 57	4 23 13	-8 26 -37	-4 72 -69	-7 89 95	0 80 -74	-8 105 102	-0 104 110	-1 150-145	L F08 FCA	0 56 -63
8 148 147	0 32 -31	5 12 6	-1 132 110	-4 88 77	J 31 - 34	-6 25 31	-2 104 93	-5 45 48	2 13 -11	-4 280 304	-4 10 24	1 59 -63	-3 21 +12	2 67 -67
9 16 -5	1 21 32	s 58 - 53	0 75 -71	-3 44 -36	H,K= 9, 11	-5 211-215	-1 114-108	-4 161 151	3 133-133	-2 22 20	-3 69 70	2 50 56	-2 16 -5	3 73 -08
	2 69 72		1 91-101	-2 43 -45	L FOB FCA	-4 65 61	0 23 -17	-3 113-102	4 49 -53	0 283 283	-2 32 -19	3 116 110	-1 18 9	
L FOR FCA	5 150-157	1 FOL FCA	3 55 -57	0 161-153	-4 70 72	-2 87 -95	2 63 -54	-2 U -2U	5 73 - 77	4 70 - 70	-1 150 146	4 44 - 34 5 20 - 31	0 70 65	H,K= 14, 6
-10 26 19	5 57 - 56	-6 47 58	4 28 3 290	1 30 36	-3 16 26	-1 23 22	3 64 96	0 44 -51	7 17 -4	6 103 104	1 0 -8	5 76 -74	2 09 -03	-3 75 79
~9 110 113	6 22 -10	-5 46 -37	5 16 -9	2 161 163	-2 16 14	0 35 30	4 50 50	1 48 60			2 153 148		3 38 44	-2 116 125
-7 69 -63	8 55 50	-3 29 -34	7 16 15	5 25 30 4 11 5	0 22 -25	2 14 -15	5 23 24 6 61 57	2 157 128	H , K = 11 , 6	1 FCB FCA	3 120 120	1 505 675	H.F. 13 .	-1 b -15
-6 12 -4		-2 60 -63	8 24 11	5 92 -91	1 100 -96	3 50 -44		4 33 38	-7 30 -12	-8 101 -98	5 64 63	-7 61 -70	L FUB FCA	1 10 2
-5 240 239	h+K= 6+ 7	-1 39 -34	9 47 - 57	6 76 -72	2 46 -42	4 105-104	H:K= 10, 8	5 0 -4	-6 48 -54	-7 8 -13	6 O 2	-0 28 -26	~3 31 34	2 23 13
-4 18 -11	-9 34 -35	0 42 -42	W-X- 0. 3	1 41 48	5 55 - 54	5 19 14	-Z O -A	5 55 -49	-5 /6 /2	-6 21 20	b. K = 1.2 - c	-5 71 75	-2 43 56	
-2 95 -77	-8 14 44	2 91 - 93	L FOB FCA	0 11 50		7 108-110	-6 17 -25	. 105 105	-3 67 -78	~4 103-103	L FOD FCA	-3 54 - 00	0 -27 -29	L FCB FCA
-1 147 141	-7 59 -63	3 39 -43	-9 17 23	н,к. 9. 7	H,K= 9, 12	8 22 -15	-5 44 -49	H,K= 11, 2	-2 76 -80	-3 146-147	-0 20 -13	-2 56 -59	1 74 74	-4 90-100
0 67 60	-6 80 -83	4 87 - 93	-8 42 -42	L FU8 FCA	L FUB FCA	H. K. 10. A	-4 72 69	L FUG FCA	-1 74 74	-2 63 -69	-5 27 -26	-1 192-192	N. K 14 /	-2 141-136
2 74 -90	-4 193-196	5 52 -16	-6 120 120	-7 62 -65	-2 22 -16	L FOB FCA	-2 242 234	-7 55 64	1 20 -9	0 126+109	-3 164 109	1 12 11	L E06 E64	2 0 -12
3 46 62	-3 107-101	H,K= 0, 12	~5 20 10	-6 72 -71	-1 49 -38	-9 121-107	-1 50 38	-0 32 -19	c 60 60	1 118 117	-2 139 145	2 20 -2	-4 89	
4 10 -2	-2 263 264	L FOB FCA	-4 190 190	-5 26 21	0 107 113	-8 22 39	0 44 29	-5 114 116	3 69 90	2 48 - 50	-1 66 -64	3 36 35	-2 135 150	H,K= 15, 1
5 158 164	0 11 29	-4 46 -30	-2 55 59	-3 142 138	2 11 13	-6 158 160	2 100 93	-4 18 -8	5 105 Lub	4 71 - 68	1 51 - 54	5 132-134	2 60 -77	L FUB FCA
7 24 35	1 163 169	-2 76 81	-1 67 -73	-2 157-153		-5 21 26	3 214 214	-2 63 - 66	0 40 41	5 94 - 57	2 110 114	/ 151 154	4 25 23	-3 113 120
8 23 -16	2 207-216	-1 10 2	0 83 90	-1 136-134	H,K= 10, 0	-4 47 38	4 127 133	-1 84 74		6 87 -90	3 49 -46	H,K= 13, 3		-2 52 -45
V 112 113	3 6 15	1 32 -19	2 59 55	1 74 74	L FOB FCA	-3 116-107	5 36 39	0 116 117	1 508 51 A	7 91 85	5 13 13	-D 97 -94	H,K# 14, 1	-1 22 -17
H,K= 8, 4	5 48 42	2 55 55	3 101 99	2 129-137	-6 37 27	-1 125-107	0,00,07	2 196-200	-6 7 -3	H.K= 12, 2		-5 24 -23	-5 63 92	1 8 14
L FOB FCA	6 109 114	3 131-135	4 9 0	3 91 03	-4 26 -a	D 23J 223	H,K= 10, 9	3 82 83	-> 03 -04	L FOB FLA	H,K= 12, 7	-4 27 35	-4 5 5	2 2o -8
-10 70 -64	A 13 20	H.K. 4. 13	5 13 -6	4 136-130	-2 102 -85	1 112-116	L FOE FCA	4 30 25	-4 40 44	-6 62 67	-5 65 62	-2 126 136	-3 43 36	N. K. 16. 2
-8 86 88	0 55 60	L FOB FCA	7 22 39	6 62 65	2 85 -85	3 22 -10	-5 101 102	6 62 59	-2 176 109	-5 51 52	-4 55 -54	-1 99 -97	-1 62 -52	L FCB FLA
-7 56 57	H,K= δ, β	-2 96 -99	8 55 67	7 44 -44	4 108 179	4 170 165	-4 39 36	7 76 75	-1 71 73	-4 203 218	-3 40 -47	0 51 - 57	0 32 - 33	-3 24 -21
-5 282 293	-8 93 6L	-1 50 53	9 19 20	н.к. 9. а	6 120 118 8 154 145	5 172-180	-3 30 17	H.K. 11. 3	0 106 104	-2 36 39	-1 95 -94	2 26 - 19	2 14 -12	-2 54 -59
-4 59 -43	-7 21 6	1 9 - 24	H,K= 9, 4	L FUB FLA	0 17 1 147	7 41 -48	~1 106+111	L FUB FCA	2 35 - 37	-1 33 49	0 29 14	3 13 10	3 74 -61	0 56 52
-3 63 -62	-6 120 122		L FUB FCA	-7 15 -22	H,K= 10, 1	8 68 55	0 64 -61	-8 66 -85	3 34 34	0.50 34	1 67 -07	4 31 37	4 63 61	1 61 -85
-1 235 220	-5 47 -44	1 FOL FCA	-8 47 -46	-5 46 -66	-9 33 30	H.K. 10. 5	2 31 -15	-7 119 122	4 41 32 5 54 -57	2 241 234	2 82 -57	5 20 6	H.K. 14. 2	2 26 14
0 364 364	-3 80 93	-10 36 41	-7 150 147	-4 26 -32	-6 50 -52	L FOB FCA	3 55 -61	-5 29 -27	6 130 134	3 82 -66	4 85 - 91	H,K* 13, 4	L FOB FCA	H,K= 15, 3
1 161 171	-2 291 284	-8 52 40	-6 10 o	-3 33 28	-7 88 82	-8 27 36	4 46 -37	-4 58 59	11 K- 11 - 0	4 29 23	W K = 12 0	L FDE FCA	-5 76 -71	L FOB FCA
3 21 -18	0 71 61	-4 121-124	-4 12 -66	-1 41 -41	-5 64 -69	~6 12 4	5 126-158	-2 71 67	L FUE FLA	6 61 73	L FD8 FCA	-5 39 29	-3 14 -17	-2 85 84
4 109-100	1 67 -63	-2 324 292	-3 45 51	0 104 86	-4 144 137	-5 213-227	H,K= 10, 10	-1 43 36	-6 0 -4		-5 99-112	-4 37 -30	-2 211 217	~1 14 11
5 17 -15	2 392 385	0 111-119	-2 127-124	1 7 12	-3 145 141	-4 39 -35	L FOB FCA	0 229-226	-> 18 -21	H,K= 12, 3	-4 17 -70	-3 43 37	-1 41 -34	0 40 48
7 113 114	3 2 -7	2 75 -83	0 129-130	2 24 -11	-2 18 14	~3 113 113	-5 81 -84	2 37 30	-4 50 55	-7 22 -12	-2 39 38	-2 24 -26	1 65 -66	2 64 60
B 157 169	5 16 -12	0 57 -45	1 17 75	4 49 52	0 204 193	-1 34 28	-3 143-150	3 13 -21	-2 37 -35	-6 22 -5	-1 63 -63	Ú 40 -45	2 64 60	2 04 -05
9 93 -91	6 58 54	έ <b>€</b> − 58 5	2 43 41	5 15 -40	1 26 23	0 47 44	-2 23 -9	4 44 34	-1 66 62	-5 16 -3	0 56 70	1 91 85	3 45 -49	H,K=_15, 4
H.K. B. 5	7 35 - 33	H.K. G. 1	3 95 -69	6 7 -18	2 229 232	1 117-109	-1 14 -21	5 30 -25	0 57 54	-3 140-135	2 35 38	2 62 61	4 127 144	1 FUB FUA
L FDE FCA	H.K= 6, 9	L FUE FCA	5 71 73	/ 0 29	4 124 122	3 54 54	1 95-104	7 105 109	2 109-137	-2 28 -22	3 74 - 76	4 60 -62	H.K= 14. 3	-1 21 -10
-9 33 -31	L FUB FCA	-10 72 -60	6 179-179	H,K= 9, 9	5 16 6	4 61 -46	2 77 -67		3 66 -71	-1 59 55	4 75 - 71	5 18 -8	L FUB FCA	0 96 104
~8 133 137	-7 45 48	-9 24 - 24	7 0 6	L FOB FCA	6 43 47	5 69 77	3 4 -14	H,K= 11, 4	4 19 -7	0 41 - 39	6.Kx 12. 0	H.K. 13. 5	-5 42 34	1 29 -31
-1 21 15	-5 23 28	-7 59 67	a 111 107	-1 23 -23 -6 63 67	a 121 119	0 34 -23 7 0 -b	4 150 130		2 62 84	2 103-101	L FOL FCA	L FOD FCA	~3 57 54	
-5-59 66	-4 26 34	-0 00 -50	H,K= 9, 5	-5 37 -48			H,K= 10, 11	-7 64 63	H,K= 11, 9	3 85 - 94	-3 33 -19	-5 122 125	-2 190 195	
-4 17 -79	-3 97 -99	-5 16 5	L FD8 FCA	-4 .0 14	H.K= 10, 2	H,K= 10, 6	L FOL FCA	-6 44 46	L FOB FLA	4 59 -72	-2 53 42	-4 34 -32	-1 63 67	
-2 82 74	-1 24 -19	-4 22 3 -3 143-156	-8 115-112	-2 73 -75	-9 49 -50	-8 74 BI	-3 55 54	-0 68 65 -4 96 100	-9 19 -35	6 14 -9	0 62 74	-3 30 15	1 44 33	
-1 68 -68	0 25 13	~2 226-234	-7 219-220	-1 56 -56	-8 25 26	-7 58 -70	-1 10 -1	-3 27 -21	-3 27 -4		1 84 91	-1 171 174	2 19 9	
0 17 5	1 50 55	-1 15 -21	-6 73 73	0 17 -3	-7 35 33	-6 61 64	0 19 -7	-2 29 -23	-2 20 14	H,K* 12, 4	2 65 60	0 49 43	3 0 -29	
1 61 33	2 64 65	0 151-135	-5 AD 17	1 11 -4	-6 185 191		1 67 66		EL 114 LUS	L FUO FLA		1 60 85	4 114 125	

TABLE II (Continued)



Figure 2.—The crystal structure of  $N(CH_3)_4[(B_9C_2H_8Br_3)_2Co]$  compared with that of KHF<sub>2</sub>. In both drawings, the cations are at z  $z = \pm 1/4$  and the anions are at z = 0. The anions at z = 1/2 (not shown) are related to those shown by the *c* glide which runs horizontally across the center of each drawing.

equivalent and amount to  $\sim 3^{\circ}$  and  $\sim 0.07^{\circ}$  Å, respectively. This is interpreted as the effect of the Br(5) of each dicarbollyl being crowded up against the hydrogens on the carbons of the other. This interpretation is supported by the short Br(5)-Br(9) and Br(5)-Br(10)distances (Table IV). If we assume that each hydrogen is 1.10 Å from its carbon and in line with the car-

bon and the opposite icosahedral vertex, the brominehydrogen distance is only 2.75 Å, which is 0.40 Å less than the sum of their van der Waals radii (1.95 + 1.2 =3.15 Å).23 A similar steric effect of about the same size, but in the opposite direction (carbons farther from

(23) L. Pauling, "The Nature of the Chemical Bond," 3rd ed, Cornell University Press, Ithaca, N. Y., 1960, p 260.

	TABLE III
DISTANCES <sup>a</sup> IN	$N(CH_3)_4[(B_9C_2H_8Br_3)_2Co]$

	Ion no						
	1	2	, 3	4			
Atoms		———Dista	nce, A				
Co-C(2)	$2.04(2)^{b}$	2.03(2)	1.98(2)	2.05(2)			
Co-C(3)	1.99(2)	2.02(2)	2.00(2)	2.09(2)			
	Rms	scatter <sup><math>c</math></sup> = 0.	034				
Co-B(4)	2.09(3)	2.13(3)	2.14(3)	2.17(3)			
Co-B(6)	2.05(2)	2.10(2)	2.12(3)	2.12(3)			
	Rms	scatter $= 0.4$	037				
Co-B(5)	2.17(3)	2.17(2)	2.15(2)	2.16(3)			
	Rms	scatter $= 0.0$	007				
Co-Br(5)	3.527(3)	3.530(3)	3.511(3)	3.527(3)			
	Rms	scatter $= 0.0$	009				
Br(5) - Br(9)	3.716(5)	3.775(4)	3.709(4)	3.725(4)			
Br(5) - Br(10)	3.689(4)	3.730(4)	3.756(4)	3.719(4)			
	Rms	scatter = $0.0$	027				
Br(9) - Br(10)	3,921(5)	3.869(5)	3,816(5)	3.876(4)			
	Rms	scatter $= 0.0$	043				
B(5)-Br(5)	1.95(3)	2.00(2)	1.96(2)	1.94(3)			
B(9) = Br(9)	1.96(3)	1.95(3)	1.98(3)	1.97(3)			
B(10) = Br(0)	1.00(0) 1.06(3)	1.08(3)	1.00(0) 1.96(3)	1.06(2)			
D(10) D1(10)	1.00 (0) Rms	scatter $= 0$	117	1.00(2)			
C(2) - C(3)	1 68 (3)	1.65(2)	1 62 (2)	1 66 (3)			
C(2) = C(3)	1,00(0) Dmc	1.00(0)	193	1.00(0)			
$C(9) \mathbf{P}(6)$	1 69 (2)	1.75(2)	$\frac{1}{1}$ $\frac{77}{4}$	1 79 (2)			
C(2) = B(0)	1.03(3) 1.64(2)	1.69(3)	1.77(4) 1.79(2)	1.70(0) 1.94(0)			
C(3) = B(4)	1.04(3)	1.08(3)	1.72(3)	1.84(3) 1.79(3)			
C(2) - B(11)	1.63(4)	1.75(4)	1.84(4)	1.72(3)			
C(3) - B(8)	1.75(3)	1.69 (4)	1.68(4)	1.64(3)			
C(2)-B(7)	1.71(4)	1.71(4)	1.86(4)	1.71(4)			
C(3)-B(7)	1.76(4)	1.73(4)	1.73(4)	1.69(4)			
- (1) - (1)	Rms	scatter $= 0.0$	J64	1 01 (1)			
B(4) - B(5)	1.91(4)	1.82(4)	1.88(3)	1.81(4)			
B(4) - B(8)	1.79(4)	1.73(4)	1.76(4)	1.82(4)			
B(4) - B(9)	1.84(4)	1.79(4)	1.88(3)	1.77(4)			
B(5)-B(6)	1.69(3)	1.78(3)	1.80(4)	1.81(4)			
B(5)-B(9)	1.81(4)	1.70(4)	1.82(3)	1.77(4)			
B(5)-B(10)	1.74(4)	1.74(4)	1.77(3)	1.81(4)			
B(6)-B(10)	1.74(4)	1.75(4)	1.81(4)	1.79(3)			
B(6)-B(11)	1.74(4)	1.84(4)	1.90(4)	1.88(4)			
B(7)-B(8)	1.88(4)	1.83(4)	1.63(5)	1.81(4)			
B(7)-B(11)	1.79(5)	1.73(4)	1.82(5)	1.77(4)			
B(7)-B(12)	1.91(5)	1.84(5)	1.74(4)	1.83(4)			
B(8) - B(9)	1.75(4)	1.79(4)	1.74(4)	1,83(4)			
B(8) - B(12)	1.89(4)	1.82(5)	1.67(4)	1.84(4)			
B(9) - B(10)	1.76(4)	1,76(4)	1.71(4)	1.75(4)			
B(9) - B(12)	1.75(5)	1.77(4)	1.62(4)	1.79(4)			
B(10)-B(11)	1.86(4)	1.76(4)	1.79(4)	1.76(4)			
B(10)-B(12)	1.71(4)	1.74(4)	1.74(4)	1.78(4)			
B(11) - B(12)	1.82(5)	1.79(4)	1.76(4)	1.79(4)			
~ (~~) **(***)	Rms	scatter = $0.0$	060				
$N-CH_{s}(1)$	1.49(5)	1.52(6)					
$N-CH_{2}(2)$	1 46(4)	1 43(7)					
$N-CH_{2}(3)$	1 49(3)	1 44(5)					
$N-CH_{0}(4)$	1.57(0)	1.53(5)					
T/-CI13(4)	1.07 (T) Dmc	scatter $- \Omega$	148				
	Ruis	scaller $= 0.0$	010				

<sup>*a*</sup> None of these distances is corrected for thermal motion. <sup>*b*</sup> The numbers in parentheses are the standard deviations in the least significant digits as calculated from the standard deviations of coordinates (Table I). <sup>*a*</sup> Each of the "rms scatter" values given is the root-mean-square deviation from the average,  $[\Sigma\Delta^2/(n-1)]^{1/2}$ , for the preceding set of distances. These are given for comparison with the standard deviations calculated from the standard deviations of coordinates.

	TABLE IV								
Average Distances <sup>a</sup> in $N(CH_3)_4[(B_9C_2H_8Br_3)_2Co]$									
No. averaged	Atoms	Distances, Å							
4	CC	$1.654 \pm 0.017^{b}$							
24	CB	$1.725\pm0.013$							
72	B–B	$1.786\pm0.007$							
12	B–Br	$1.963 \pm 0.008$							
8	Co-C(2,3)	$2.024 \pm 0.013$							
8	Co-B(4,6)	$2.115 \pm 0.014$							
4	Co-B(5)	$2.162 \pm 0.014$							
4	Co-Br(5)	$3.524 \pm 0.005$							
8	Br(5)-Br(9,10)	$3.727 \pm 0.010$							
4	Br(9)-Br(10)	$3.870\pm0.025$							
8	N-CH3	$1.491 \pm 0.018$							

<sup>*a*</sup> None of these distances is corrected for thermal motion. <sup>*b*</sup> The error figures are our best estimate of the precision of these averages. They have been pessimistically chosen as the greater of  $[(\Sigma \Delta^2)^{1/2}/(n-1)]$  or  $[(\Sigma \sigma^2)^{1/2}/n]$ , where  $\Delta$  is the deviation from the average,  $\sigma$  is the standard deviation of a single distance as calculated from the standard deviations of coordinates, and *n* is the number of distances averaged. The latter expression was the larger for the C–C, B–Br, and Co–B( $\tilde{\sigma}$ ) distances.

metal than the borons) was found<sup>24</sup> in  $B_9C_2H_9(CH_3)_2$ -PdC<sub>4</sub>(C<sub>6</sub>H<sub>5</sub>)<sub>4</sub> in which the methyl groups are on the dicarbollyl carbons. These steric effects are not to be confused with the larger shift (about 0.6 Å) observed by Wing<sup>25</sup> in the Cu(B<sub>9</sub>C<sub>2</sub>H<sub>11</sub>)<sub>2</sub><sup>2-</sup> anion where bonding effects are believed to be involved.

The electrophilic bromination sites found for this anion may be rationalized if we say that the charge distribution in the dicarbollyl portion is similar to that in 1,2-dicarbaclovododecaborane  $(B_{10}C_2H_{12})$ , but modified by a polarization induced by the Co(III). Substitution sites<sup>19-21</sup> and dipole moments<sup>26</sup> found for B10C2H12 derivatives and molecular orbital calculations<sup>19,20</sup> for the parent compound all lead to the conclusion that the borons farthest from carbon are the most susceptible to electrophilic attack and this susceptibility decreases as the carbons are approached. This analogy, considered alone, would indicate that our B(9) and B(10) are the most subject to electrophilic attack and B(5) and B(12) are second. Polarization by the cobalt would then determine the choice between B(5) and B(12), possibly causing B(5) to become more subject to electrophilic attack than the B(9)-B(10)pair.

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<sup>(25)</sup> R. M. Wing, J. Am. Chem. Soc., 89, 5599 (1967).